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TITLE: Grinding mill for dispersed metal oxide agglomerate - has numerous milling members on several stacked dispersion permeable separating floors in housing

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ABSTRACT:

The large number of spherical milling members (86,88) are located within the housing (4) on stacked floors, permeable to the dispersion dividing floors (66, 68, 70). The abrading force is applied to the agglomerate by vibratory motion

of the housing. The sieve like dividing floors are detachably held on a central column (10) of the housing. This central column is cooled from the inside. The housing is axially formed of segments and has a detachable cover.

USE/ADVANTAGE - For magnetic recording tape mfr., with reliable agglomerate removal and homogeneous distribution of the metal oxides in the dispersion.

PATENT ABSTRACTS OF JAPAN

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(54) MAGNETIC FLUID OF SILICONE OIL BASE AND ITS MANUFACTURE

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain magnetic fluid excellent in dispersion, fluidity and endurance, by making the fluid contain silicone oil, carboxyl group containing anion based surface-active agent, and magnetic metal oxide particles and/or magnetic metal particles.

SOLUTION: The title magnetic fluid of silicone oil base contains silicone oil, carboxyl group containing anion based surface-active agent, and magnetic metal oxide particles and/or magnetic metal particles. The magnetic fluid is manufactured as follows. Alkaline suspension is obtained by adding alkaline agent to suspension liquid dispersing magnetic metal oxide particles and/or magnetic metal particles; carboxyl group containing anion based surface active agent to the alkaline suspension; pH of the suspension obtained in the above is prepared to be at most 2; the obtained acid suspension liquid is dried; and the dried object and silicone oil are mixed. Thereby magnetic fluid of low vapor pressure which is applicable to a wide temperature range, non-toxic and safe can be obtained.

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CLAIMS

[Claim(s)]
[Claim 1] The magnetic fluid of the silicone oil base characterized by containing silicone oil, a carboxyl group content anion system surfactant, a magnetic metallic-oxide particle, and/or magnetic metal particles.
[Claim 2] The magnetic fluid of said silicone oil base according to claim 1 which is the compound which has a carboxyl group through the alkylene group whose carbon numbers are 1-7 at the end of the polyoxyalkylene group with which said carboxylic-acid radical content anion system surfactant combines an alkyl group through ether linkage.
[Claim 3] The process of the magnetic fluid of the silicone oil base which obtains alkaline suspension by adding alkali chemicals to the suspension which distributes a magnetic metallic-oxide particle and/or magnetic metal particles, adds a carboxyl group content surfactant to this alkaline suspension, prepares pH of the suspension obtained after that or less to two, dries the acid suspension obtained, and is characterized by mixing the dry matter obtained and silicone oil.
[Claim 4] The process of the magnetic fluid of the silicone oil base which adds alcohols to said dry matter according to claim 3, and is characterized by mixing and heating the black gel object and silicone oil to generate.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] About the magnetic fluid of the silicone oil base, and its process, in more detail, this invention is excellent in dispersibility, a fluidity, and endurance, and relates to the magnetic fluid of the silicone oil base which can moreover be suitably used in the temperature field of the large range from ordinary temperature, and its process.
[0002]
[Description of the Prior Art] A magnetic fluid makes it come to distribute the particle of the very detailed ferromagnetic whose particle size is about 10nm in the condition of having been stabilized in the inside of a solvent, for example, an organic solvent, or underwater. Since flocking settling of a particle does not happen in a gravity field, or the usual centrifugal field or a magnetic field, a magnetic fluid is served like the liquid which has ferromagnetism. the attempt of new application , such as the sealing operation acquire by this property not have a parallel in other ingredients , for example , put a magnetic fluid on the concentration part of magnetic flux , gravity concentration using the magnetic buoyancy commit to the nonmagnetic matter in the magnetic fluid put on the bottom of a field gradient or a storage ingredient using magnetism , magnetic ink for printers , a waste oil treatment , and various devices , be propose , and application be the ingredient expect widely .
[0003] About the process of such a ferromagnetic, for example to JP,53-17118,B A complement is added. the ferromagnetic oxide impalpable powder by "wet method -- using -- the water suspension -- the basic salt of unsaturated fatty acid -- the impalpable powder in this suspension -- the above-mentioned fatty-acid ion -- a monolayer absorption layer is completed at least -- making -- This fatty-acid ion is made to stick to the impalpable powder front face in the above-mentioned suspension more than a single molecule, and the manufacture approach of the magnetic fluid characterized by adding an acid, making with the aqueous acids of pH 7-5, making a suspensoid condense, filtering this aggregate, washing, dehydrating, and making it distribute in oil" is indicated after that. It is understood that this manufacture approach is the manufacture approach of the magnetic fluid of the oil base.
[0004] Here, although the dispersion medium of the magnetic fluid named oil generically contains various oil, such as hydrocarbons, such as paraffin which is petroleum products, the hydride or the compounded hydrocarbon, ester, polyglycol, a polyphenyl ether, halocarbon, and chloro FURUORU carbon, a magnetic particle does not show good dispersibility to all these oil.
[0005] A dispersion medium is chosen according to the property required of a magnetic fluid. For example, although a magnetic fluid is a magnetic material which has a fluidity and many of applications use this description, the viscosity of the magnetic fluid which governs a fluidity is influenced by the viscosity of the oil first used in the first place as a dispersion medium. And the viscosity of this oil changes with temperature. Moreover, it is the factor in which evaporation of oil also spoils a fluidity. In the application using a fluidity, in the presentation which constitutes a magnetic fluid, first, viscosity of a dispersion medium must be low and you must be oil which cannot evaporate easily. And in order to bring the magnetic fluid into a large temperature requirement and to operate to stability, it is desirable for there to be as much as possible little viscous change according to temperature in own thermal resistance and cold resistance of oil. Although this property is shown as a viscosity index, many cannot evaporate easily due to low viscosity, a viscosity index is known as best oil in the top where thermal resistance and cold resistance are also high, and silicone oil (organic silicone) has little viscous change accompanying a temperature change. Moreover, although a dispersion medium must be physiologically safe when using a magnetic fluid for a remedy, cosmetics, etc., it is known that many of silicone oil is inactive physiologically. However, this silicone oil was one of the oil which distribution of a magnetic particle does not distribute easily.
[0006] "The low-temperature-service magnetic fluid which a dispersion medium is a specific siloxane compound and is

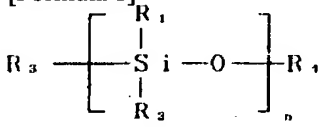
characterized by a surfactant consisting of the 1st surfactant which is the hydrocarbon compound shown by general formula R-X (however, R a hydrocarbon group and X polar groups, such as a carboxyl group and a sulfone radical), and the 2nd surfactant which is a specific siloxane compound in the magnetic fluid which makes it come to distribute the ferromagnetic particle which made the surfactant adsorb into a dispersion medium" is indicated by JP,63-175401,A.

[0007] JP,54-40069,B "to the water suspension of the ferromagnetic oxide impalpable powder by the wet method Complement addition is carried out. the basic salt of oleic acid, or the basic salt of linolic acid -- the impalpable powder in this suspension -- oleic acid ion or linolic acid ion -- a monolayer absorption layer is completed at least -- making -- After making oleic acid ion or linolic acid ion stick to the impalpable powder front face in the above-mentioned suspension more than a single molecule, Filter, after adjusting so that this liquid may turn into a less than seven-PH acidic solution, and a powder particle is obtained. After washing this powder particle using water or other polar solvents, it sets underwater and the manufacturing method of the magnetic fluid with which the carbon number of a hydrocarbon chain made the dispersion medium the water characterized by adding nine or more anion molds or a non-ion mold surfactant, and making with suspension" is indicated. It is understood that this manufacture approach is a process of a water base magnetic fluid.
[0008] "JP,4-108898,A In the magnetic fluid constituent which comes to distribute the dispersion medium which uses a silicone oil as a principal component, a ferromagnetic particle, and this ferromagnetic particle in said dispersion medium The coupling agent equipped with the functional group which reacts with the functional group of said ferromagnetic particle front face, and carries out a direct chemical bond to the ferromagnetic particle front face concerned through the resultant at this time, The magnetic fluid constituent characterized by making said dispersion medium distribute said ferromagnetic particle through the reactant silicone oil equipped with other functional groups of the coupling agent concerned and the functional group which carries out a direct chemical bond" is indicated.
[0009] The similar constituent is proposed also in JP,63-213326,A. However, since these constituents must prepare a two-layer adsorption layer using two sorts of adsorbents to the magnetic particle which is a dispersoid, a presentation and its process are complicated, when an adsorption layer becomes thick, viscosity becomes high, and the description of the silicone oil which is low viscosity cannot be employed efficiently.
[0010]
[Problem(s) to be Solved by the Invention] The object of this invention is excellent in dispersibility, a fluidity, and endurance, and is to offer [it being suitably used in the temperature field of the large range from ordinary temperature moreover, and offering the magnetic fluid of the silicone oil base where a viscosity index is low, and] the easy process of the magnetic fluid of the silicone oil base which was excellent in this way.
[0011] Furthermore, the object of this invention is low vapor pressure, and is to offer the magnetic fluid of the safe silicone oil base, and its process by avirulent.
[0012]
[Means for Solving the Problem] Invention of a publication to this application claim 1 for solving said technical problem It is the magnetic fluid of the silicone oil base characterized by containing silicone oil, a carboxyl group content anion system surfactant, a magnetic metallic-oxide particle, and/or magnetic metal particles. Invention according to claim 2 said carboxylic-acid radical content anion system surfactant At the end of the polyoxyalkylene group which combines an alkyl group through ether linkage A carbon number is the magnetic fluid of said silicone oil base according to claim 1 which is the compound which has carboxyl group ** through the alkylene group which are 1-7. Invention according to claim 3 Alkaline suspension is obtained by adding alkali chemicals to the suspension which distributes a magnetic metallic-oxide particle and/or magnetic metal particles. Add a carboxyl group content surfactant to this alkaline suspension, and pH of the suspension obtained after that is prepared or less to two. It is the process of the magnetic fluid of the silicone oil base characterized by drying the acid suspension obtained and mixing the dry matter obtained and silicone oil. Invention according to claim 4 Alcohols are added to said dry matter according to claim 3, and it is the process of the magnetic fluid of the silicone oil base characterized by mixing and heating the black gel object and silicone oil to generate.
[0013]
[Embodiment of the Invention] Hereafter, the magnetic fluid of the silicone oil base of this invention and its process are explained to a detail.
[0014] <Magnetic fluid of the silicone oil base> The magnetic fluid of the silicone oil base of this invention contains silicone oil, a surfactant, a magnetic metallic-oxide particle, and/or magnetic metal particles.
[0015] - Silicone oil - The silicone oil in this invention is a base oil used as a dispersion medium, and especially as long as it is a liquid in ordinary temperature, it does not have a limit. In the point that cold resistance, low volatility, and

chemical stability are excellent as suitable silicone oil in this invention, the silicone compound shown by the following general formulas (** 1) is mentioned.

[0016]

[Formula 1]



[0017] (However, R1, R2, R3, and each R4 show an aliphatic hydrocarbon radical, an alicycle group hydrocarbon group, and an aromatic hydrocarbon radical.) R1, R2, R3, and R4 Different, even if mutually the same.

As silicone oil used for this invention, dimethyl silicone oil, methil hydrogen polysiloxane, methylphenyl silicone oil, alpha-methyl-styrene denaturation silicone oil, alkyl denaturation silicone oil, alcoholic denaturation silicone oil, amino denaturation silicone oil, polyether denaturation silicone oil, the halo silicone which it chlorinates or comes to fluorinate can be mentioned, for example. One desirable also in these can mention methylphenyl polysiloxanes, such as tetra-phenyl tetramethyl trisiloxane and PENTA phenyl trimethyltrisiloxane, etc.

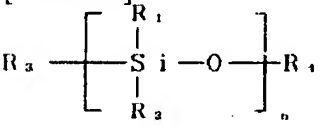
[0018] As for the silicone oil used for this invention, it is desirable that the viscosity in those 25 degrees C is 10-1,000cSt. Moreover, as for silicone oil, what has the specific gravity within the limits of 0.94-1.11 is desirable. Such a methylphenyl polysiloxane is physiologically [viscosity is also low and] safe. For example, it is more nearly available than Shin-Etsu Chemical Co., Ltd. as "KF-50" (trade name), "KF-53" (trade name), "KF-54" (trade name), and "KF-56" (trade name).

[0019] - Surface active agent - The surface active agent used for this invention can mention the anionic surfactant which there will be no limit especially if it is a carboxyl group content anion system surface active agent and is the surface active agent which has a carboxyl group in a molecule, for example, contains a carboxyl group, and the amphoteric surface active agent containing a carboxyl group.

[0020] As a carboxyl group content anion system surface active agent, a carboxylic acid or its salt, an alkyl lactate, N-acylamino acid chloride, an alkyl ether carboxylic acid, or its salt can be mentioned. An alkyl ether carboxylic acid or its salt is still more desirable also in these, the compound which has a carboxyl group through the alkylene group whose carbon numbers are 1-7 at the end of the polyoxyalkylene group which divides also in it and combines an alkyl group through ether linkage is desirable, and the compound which has a carboxyl group through the alkylene group whose carbon numbers are 1-3 at the end of the polyoxyalkylene group which combines an alkyl group especially through ether linkage is desirable. The following formulas (** 1) can show the compound which has a carboxyl group through the alkylene group whose carbon numbers are 1-7 at the end of the polyoxyalkylene group which combines an alkyl group through this ether linkage.

[0021]

[Formula 1]



[0022] R1-O-(CH2 CHR2-O-) n-(CH2) m-COOX R1 [however,] -- an alkyl group and the alkyl group whose carbon numbers are 5-30 preferably -- Still more preferably, a carbon number is the alkyl group which are 10-24, and it is R2. They are a hydrogen atom or a methyl group. n is an integer, is the integer of 1-30 preferably, and is the integer of 2-10 still more preferably. the alkyl group which m is an alkyl group whose carbon numbers are 1-7, and are 1-3 preferably -- it is -- further -- desirable -- 1 -- it is -- X -- alkali metal, such as a hydrogen atom, or Na, K, or -NH3 it is .

The carboxyl group content surfactant in this invention is usually used as a water solution. When a carboxyl group content surfactant is used as a water solution, sufficient solubility is desired to silicone oil. When silicone oil is a methylphenyl polysiloxane, the sodium salt of the polyoxyethylene tridecyl ether acetic acid in which solubility high as a carboxy group content surface active agent is shown is desirable.

[0023] - A magnetic metallic-oxide particle and magnetic metal particles - The magnetic metallic-oxide particle in this invention is expressed with the following empirical formulas.

[0024]

(MgO) X, Y, and Z fill the following relation among X Y (ZnO) Z (Fe 2O3), however a formula.

[0025]
0.2 A unit ferrite and a restoration ferrite are mentioned as a magnetic metallic-oxide particle expressed with <=X<=1.0, 0.9<=Z<=1.1, and the X+Y= 1 aforementioned empirical formula.
[0026] The presentation of these ferrites is set up according to the property required of a magnetic fluid. For example, when high magnetization is called for, Fe ferrite and a black color tone are emphasized and change of the magnetization by Mn ferrite and temperature is used, a Mn-Zn ferrite etc. is desirable.
[0027] Particles, such as Fe, Co, nickel, etc. which are obtained as magnetic metal particles in this invention by the reduction in liquid, the vacuum evaporation process, the spark erosion method, the thermal decomposition method, a plasma-CVD method, etc., can be mentioned.
[0028] In this invention, either said magnetic metallic-oxide particle and magnetic metal particles can be used as a magnetic component, and the mixture of a magnetic metallic-oxide particle and magnetic metal particles can also be used as a magnetic component.
[0029] - As concentration of the magnetic metallic-oxide particle distributed by the presentation-silicone oil of the magnetic fluid of the silicone oil base, and/or magnetic metal particles, as long as a fluidity is permitted, it can be set as arbitration. Since magnetization of the magnetic fluid itself increases, magnetic properties will improve and viscosity will also increase if concentration of a magnetic metallic oxide and/or magnetic metal particles is made high, a fluidity falls. Moreover, the upper limit of the concentration of the magnetic metallic-oxide particle which shows sufficient fluidity, and/or magnetic metal particles is usually 60 % of the weight. If the magnetic fluid which has magnetic properties and viscosity within practical limits is obtained, the concentration of a magnetic metallic-oxide particle and/or magnetic metal particles will usually be 6 - 40 % of the weight. Moreover, silicone oil is 30 - 91 % of the weight preferably 20 to 92% of the weight. A carboxyl group content surfactant is 3 - 20 % of the weight preferably two to 40% of the weight.
[0030] If said silicone oil and surfactant, a magnetic metallic-oxide particle, and/or magnetic metal particles are in said within the limits, the object of this invention can be further attained now, and the silicone oil base magnetic fluid which is a final product will be excellent in dispersibility, a fluidity, and endurance, and, specifically, moreover, can come to be suitably used in the temperature field of the large range from ordinary temperature.
[0031] In addition, the magnetic fluid of the silicone oil base of this invention can also make various kinds of additives contain, as long as it is the range which does not check the object of this invention in order to raise further the property of the magnetic fluid of the silicone oil base of this invention, or in order to give a new property in addition to the original property of the magnetic fluid of the silicone oil base of this invention.
[0032] As said additive, a conductive grant agent, an antioxidant, an antigelling agent, a color, a pigment, a viscosity index improver, an adhesiveness-reducing agent, a thickener, a metal deactivator, electrorheological fluid, liquid crystal, other magnetic fluids, etc. can be mentioned. These are added as an emulsion, a capsule, fine particles, a polymer, and a surfactant.
[0033] - The magnetic fluid of the process-silicone oil base of the magnetic fluid of the silicone oil base can be suitably manufactured by the manufacture approach of this invention.
[0034] In the approach of this invention, the aqueous suspension of a magnetic metallic-oxide particle and/or magnetic metal particles is prepared first. A metallic-oxide particle and/or magnetic metal particles can use for and acquire the hydrothermal crystallization method using the salt of the same metal as the metal in a magnetic metallic-oxide particle and/or magnetic metal particles, or a coprecipitation method. A coprecipitation method is desirable.
[0035] The important thing in this invention is preparing this aqueous suspension to alkalinity. Aqueous suspension can be easily made into alkalinity by adding alkali chemicals, for example, caustic soda etc., to aqueous suspension. As said alkali chemicals, it is not limited to caustic soda, but especially if it is the compound which can make said aqueous suspension alkalinity, there will be no limit. As pH of the aqueous suspension prepared by alkalinity, 11-12 are suitable.
[0036] In the approach of this invention, a carboxyl group content surfactant is added to the obtained alkaline suspension. As an addition of a carboxyl group content surfactant, it is usually 20 - 30 % of the weight preferably at most 50% of the weight to the magnetic metallic oxide and/or the magnetic metal particles in alkaline suspension. If the addition of this carboxyl group content surfactant is made to increase, precipitate may be produced and it may not become a perfect dispersed system.
[0037] In addition, a bleaching powder sill radical content surfactant may add this carboxyl group content surfactant to alkaline suspension directly, without diluting this to a solvent, for example, water, and adding to alkaline suspension, and using a solvent.
[0038] Subsequently, the acid is added to the alkaline suspension of the obtained surfactant content, and the pH is made

or less into two. It is important to make pH or less into two in the approach of this invention. It is because the object of this invention cannot be well attained if pH of that liquid exceeds 2 even if it adds the acid to the alkaline suspension of surfactant content.

[0039] As acid, pH of said suspension can be made or less into two, and especially unless it moreover has an adverse effect on each component in suspension, there is no limit. A sulfuric acid, a hydrochloric acid, etc. are suitable as this acid.

[0040] Subsequently the acid suspension obtained by addition of the acid is dried. A seemingly viscous dry matter is obtained by desiccation.

[0041] Moreover, when high concentration is asked for the magnetic fluid containing a magnetic particle, it is desirable to process the thin dispersed system currently distributed thoroughly by the following approaches.

[0042] That is, this concentration approach mixes the alcohols of tales doses with the comparatively thin dispersion liquid which have not produced the precipitate which comes to add a carboxyl group content surfactant to said alkaline suspension, and its dispersion liquid mostly. Then, a black gel object generates and sediments. Sedimentation will become still more prompt if the inclination of a magnetic field is given at this time. This black gel object is once separated from a supernatant. The separation approach may be extent which samples and removes a supernatant according to conventional methods, such as centrifugal separation and filtration. Although alcohols remain in the black gelation object, silicone oil is added and agitated again and Flushing heated at 150 degrees C or less removes the alcohols which remain. At this time, the concentration of a magnetic particle can be determined as arbitration according to the amount of the silicone oil added again, and even if a magnetic particle is high concentration, it does not generate precipitate.

[0043] In addition, as said alcohols, although a methanol, ethanol, propanol, a butanol, etc. are mentioned, desirable they is a methanol and ethanol.

[0044] - Application of the magnetic fluid of the silicone oil base - The magnetic fluid of the silicone oil base concerning this invention is excellent in dispersibility, a fluidity, and endurance, and, moreover, can be suitably used in the temperature field of the large range from ordinary temperature. Since it has such an outstanding property, the magnetic fluid of the silicone oil base concerning this invention is used suitable for a sealing compound, various sensors, an actuator, lubricant, viscous damping, a load maintenance guide, a fluid drive, heat exchange, position control, a liquid-liquid seal, a bearing, a damper, a heat pipe, a heat sink, ink, cosmetics (eyeliner), etc.

[0045]

[Example]

(Example 1) The colloid of Fe ferrite was beforehand obtained with the coprecipitation method. That is, this was agitated heating the water solution which contains a ferrous sulfate and ferric sulfate by the concentration of one mol/l., respectively at 80 degrees C, under churning, 6-N sodium-hydroxide water solution warmed at 80 degrees C at this was added, and pH was set to 11.5. At this time, the dark-colored particle deposited in the solution.

[0046] It applied to the pan for 10 to 60 minutes, warming and churning were continued, and Na salt of a polyoxyethylene tridecyl ether acetic acid was added to the suspension which this particle subsequently suspended. After agitating further after addition, it cooled even to the room temperature, the 3-N sulfuric-acid water solution was added, and pH of suspension was set to 2. Consequently, Fe ferrite particle which adsorbed the polyoxyethylene tridecyl ether acetic acid condensed and sedimented.

[0047] In addition, it was made into 0, 30, 50, 70, and 100 or 120,150,170,200% of the weight of nine kinds to Fe ferrite, the addition, i.e., the amount of adsorption, of said polyoxyethylene tridecyl ether sodium acetate salt.

[0048] This aggregate was filtered, and it dehydrated, it heated and dried at 90 degrees C with the dryer, and the black gel object was obtained.

[0049] The desiccation product, i.e., said gel object, separated from water is called magnetite for short below.

[0050] said 9 kinds of magnetite 15.0g -- each was put in into the glass tube bottle and the following actuation was performed per nine kinds of samples.

[0051] 30ml of ethanol was put in into said each of glass tube bottle. After adding ethanol, the supersonic wave was applied to said each of glass tube bottle. After applying a supersonic wave, said glass tube bottle was placed on the permanent magnet, and magnetite was settled.

[0052] Since ethanol remained as supernatant liquor when magnetite was settled as mentioned above, the decantation removed this ethanol.

[0053] Washing by above-mentioned ethanol was performed 1 to 4 times.

[0054] Magnetite 2g which obtained by performing ethanol washing was dissolved in the methylphenyl polysiloxane, using the sand bath, it heated for 30 minutes, ethanol was removed, and 90 degrees C of magnetic fluids were obtained.

After cooling, when magnetization was measured, the example whose addition of a polyoxyethylene tridecyl ether sodium acetate salt is 70% as shown in drawing 1 showed the highest magnetization value.
[0055] Subsequently, the above-mentioned ethanol washing was performed 2 to 4 times to the magnetite at the time of adding a polyoxyethylene tridecyl ether sodium acetate salt 70%.
[0056] Methylphenyl silicone oil (Shin-Etsu Chemical Co., Ltd. make; KF-56) 15g was added to the solution after washing and in said glass tube bottle, and ethanol was thoroughly removed by heating using Sand Bath.
[0057] After removing ethanol thoroughly, said glass tube bottle was put on the permanent magnet, and day neglect was carried out. In addition, magnetization of the permanent magnet at this time was about 2500 gauss, as a result of measuring with a gauss meter.
[0058] The supernatant magnetic fluid after neglect and in said glass tube bottle was extracted. After extracting, the magnetic balance performed magnetization measurement for said magnetic fluid.
[0059] Drawing 2 shows the magnetization measurement result according to count of ethanol washing.
[0060] Next, 100 degrees C and 1-hour heating churning were carried out for the suspension in said glass tube bottle by Sand Bath.
[0061] After carrying out heating churning, the duplex cylinder juxtaductal type rotational viscometer performed measurement of viscosity.
[0062] Drawing 3 shows the measurement-of-viscosity result according to count of ethanol washing.
[0063] In this way, the silicone base magnetic fluid was able to be obtained. The thing with low inactive and congealing point magnetism is [the thing] excellent in size, dispersibility, a fluidity, and endurance can be mentioned good [the viscosity change by low vapor pressure and temperature / smallness and electric insulation] as a property of this silicone base magnetic fluid, and chemically.
[0064] When the viscosity change by the temperature change was especially measured using the rotational viscometer also in this, 20 degrees C viscosity change and 80-degree C viscosity change were less than 50%.
[0065] Moreover, when electric insulation was measured using the high-tension circuit tester, dielectric breakdown did not happen certainly to mm in 3kV /.
[0066] Distributed stability was stable, without condensation taking place, even if it will leave it for neglect and 30 days on a 2,500 gauss permanent magnet for one day.
[0067] (Example 2) Magnetite 15.0g before ethanol washing obtained in said example 1 and methylphenyl polysiloxane (Shin-Etsu Chemical Co., Ltd. make; KF-56) 15g were mixed, and dispersion liquid were obtained by carrying out heating churning.
[0068] Ethanol washing of the dispersion liquid was carried out by mixing and agitating this dispersion liquid and 30ml of ethanol. The supersonic wave was applied to the mixture of dispersion liquid and ethanol. After applying a supersonic wave, said mixture was placed on the permanent magnet and magnetite was settled.
[0069] Since ethanol and a methylphenyl polysiloxane remained as supernatant liquor when magnetite was settled as mentioned above, the decantation removed both.
[0070] Above-mentioned washing was performed 1 to 4 times.
[0071] 20g of slurry-like objects after said washing and methylphenyl polysiloxane (Shin-Etsu Chemical Co., Ltd. make; KF-56) 15g were added, and ethanol was thoroughly removed by heating using Sand Bath.
[0072] The silicone base magnetic fluid was obtained by removing ethanol thoroughly. This silicone base magnetic fluid was placed on the permanent magnet, and day neglect was carried out. In addition, magnetization of the permanent magnet at this time was about 2500 gauss, as a result of measuring with a gauss meter.
[0073] The supernatant magnetic fluid after neglect and in said glass tube bottle was extracted. After extracting, the magnetic balance performed magnetization measurement for said magnetic fluid.
[0074] Drawing 4 shows the magnetization measurement result according to count of ethanol washing.
[0075] After performing said magnetization measurement, the duplex cylinder juxtaductal type rotational viscometer performed measurement of viscosity.
[0076] Drawing 5 shows the measurement-of-viscosity result according to count of ethanol washing.
[0077] In this way, the silicone base magnetic fluid was able to be obtained.
[0078] (Example 3) Magnetite 60.0g obtained in said example 1 was put in into the beaker.
[0079] 120ml of ethanol was added to said beaker. After adding ethanol, the supersonic wave was applied to said beaker. After applying a supersonic wave, said beaker was placed on the permanent magnet and magnetite was settled.
[0080] Since ethanol remained as supernatant liquor when magnetite was settled as mentioned above, the ethanol which is a supernatant was removed.
[0081] Above-mentioned washing was performed 3 times.

[0082] Methylphenyl polysiloxane (Shin-Etsu Chemical Co., Ltd. make; KF-56) 60.0g was added to the solution in said beaker, and full clearance of the ethanol was carried out by heating using Sand Bath.
[0083] After carrying out full clearance of the ethanol, said beaker was put on the permanent magnet and day neglect was carried out.
[0084] The supernatant magnetic fluid after neglect and in said beaker was extracted. After extracting, the magnetic balance performed magnetization measurement for said magnetic fluid.
[0085] In addition, it was shown in drawing 6 , having used said magnetization measurement result as "concentration before."
[0086] Next, said magnetic fluid was moved to another beaker, and 39ml of ethanol was added into it. After adding ethanol, said beaker was left on the permanent magnet on the 1st, and magnetite was settled. The ethanol and the silicone oil which are a supernatant were removed after precipitation.
[0087] After removing, methylphenyl polysiloxane (Shin-Etsu Chemical Co., Ltd. make; KF-56) 19.62g was added, by heating using Sand Bath, full clearance of the ethanol was carried out and it was condensed.
[0088] The magnetic balance performed magnetization measurement for the magnetic fluid after concentration.
[0089] It was shown in drawing 6 , having used said magnetization measurement result as the "concentration back."
[0090] In this way, the silicone base magnetic fluid was able to be obtained.
[0091] (Example 4) Mixing and a supersonic wave were applied to the methylphenyl polysiloxane, and it was made to distribute the dry matter obtained in the example 1. The mixing ratios at this time were 0.2, and 1, 2 and 3 about the condensation dry matter to the methylphenyl polysiloxane 1. Every 80ml base is these dispersion liquid about 80cm, respectively 2 It put into cylindrical glassware (petri dish), and put for one month at the room temperature on the magnet with a surface inductive flux of 2,500 gauss. Although the mixing ratio was not accepted in a pars basilaris ossis occipitalis even if it made the container the upside-down in the dispersion liquid of 1:0.2, the mixing ratio made sediment the volume by the dispersion liquid of 1:1, and it had about 1/of sediment of 2.
[0092] Moreover, most was sedimenting in the dispersion liquid of 1:2 and 1:3. When the magnetization of a supernatant part in which a mixing ratio removes precipitate from the mixed liquor of 1:1, and forms the stable dispersed system was measured, in 8,000 oersted (Oe), they were 2 emu/g.
[0093] (Example 5) Magnetization of the distributed part of the mixed liquor obtained considering the mixing ratio as 1:1 in the example 4 was 2 emu/g in 8000 oersted (Oe).
[0094] This distributed part was condensed by the next actuation. 36ml of ethanol was added to 39ml of distributed parts, and also on the permanent magnet, black gel was produced and it precipitated. After inclining and removing supernatant liquor, methylphenyl polysiloxane 19.62g was added and agitated, black gel was distributed, and it heated for 30 minutes at 100 degrees C with the sand bath. The capacity of the dispersion liquid after room temperature radiationnal cooling was 30ml, and magnetization was 4.8 emu/g in 8,000 oersteds (Oe). The magnetization curve of concentration actuation before in this invention and the back was as drawing 7 .
[0095] In 30ml of these dispersion liquid, a base is 2 about 80cm. Generation of precipitate was not accepted, although it put into the petri dish and being put for one month at the room temperature on the magnet with a surface inductive flux of 2,500 gauss.
[0096] That is, by this invention, it means that the distributed part obtained in the example 4 was condensed about 1.3 times, moreover magnetization increased substantially, and the stable distributed condition was maintained in the magnetic field.
[0097] As mentioned above, the magnetic fluid of the silicone oil base excellent in dispersibility, a fluidity, and endurance was able to be obtained according to these examples.
[0098]
[Effect of the Invention] By this invention explained in full detail above, the viscosity change by low vapor pressure and temperature is small, electric insulation is good, and is inactive chemically, magnetization is size, and it excels in dispersibility, a fluidity, and endurance, and the viscosity change especially by the temperature change can be small, and the magnetic fluid of the silicone oil base which was moreover excellent in electric insulation and distributed stability can be offered.
[0099] Moreover, by this invention, the easy process of the magnetic fluid of the silicone oil base which was excellent in the property as mentioned above can be offered.

[Translation done.]